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14. ABSTRACT
The use of fugitive coatings to produce controlled, uniform interface gaps in oxide composites has been assessed. The results demonstrate that the notched and unnotched tensile properties of gap-containing materials are superior to those of materials with bonded interfaces, especially after long-term (1000 h) aging at targeted service temperatures (1200°C). The thermochemical stability of these materials represents a significant advancement toward the goal of enhanced durability. Additionally, processing principles and implementation protocols for producing 3D oxide composites with high-integrity matrices have been established. Shrinkage cracks in these matrices has been mitigated through the addition of comparatively large particles to the matrix pockets between tows. The concept has been demonstrated using SiC, although extensions to other particle types (e.g. mullite) should, in principle, be feasible. Finally, upon expanding collaborations with AFRL personnel, the efficacy of monazite coatings in the toughness and durability of 3D composites with alumina/mullite fibers has been initiated.

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DURABLE HIGH-PERFORMANCE OXIDE COMPOSITES

AFOSR GRANT NO. F49550-05-1-0134

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Objectives

The program is focused on durable oxide-based continuous-fiber ceramic composites (CFCCs). Systems of this type are being considered for use in aircraft and land-based gas turbines, as well as future expendable and reusable launch vehicles of interest to the Air Force. *Two major research thrusts are being pursued: (i) design and synthesis of composite systems with 3D fiber architectures, and (ii) development of thermochemically stable interface concepts.* The short-term goals are: (i) to identify architectures that can be manufactured using established weaving technology and produce significant improvements in through-thickness composite properties with minimal change in-plane; and (ii) assess the utility of monazite coatings on Nextel 720™ fibers. The long-term objectives include development and validation of material models that encompass phenomena operating at various length scales, ranging from microscopic (e.g. junctions of matrix particles) to mesoscopic (e.g. deformation response of fiber tows) to macroscopic (e.g. component level). A major underlying theme is the interplay between microstructural parameters, anisotropy of composite properties (matrix- vs. fiber-dominated), and long-term durability at elevated temperatures.

Collaborations

Enhancements in the scope of the program have been enabled through collaborations with investigators at AFRL and Teledyne Scientific as well as Siemens Power Generation.

Recent Developments

A preliminary assessment has been made of the efficacy of monazite coatings on Nextel 720™ fibers. Using coated bundles provided by AFRL, unidirectionally-reinforced minicomposite specimens with a somewhat porous oxide matrix have been fabricated and tested. Significant fiber pullout is evident on the resulting fracture surfaces (Figure 1),

consistent with the expectation of a weak interfacial bond between the fibers and the coatings. A more critical assessment awaits future tests on composites with nearly fully dense matrices.

A method for application of monazite coatings onto oxide fibers *after* matrix infiltration and sintering has been devised and demonstrated[1]. It employs an intermediate fugitive coating followed by impregnation and pyrolysis of a monazite precursor solution. Composites with monazite coatings produced in this manner exhibit superior mechanical performance to composites without any coating, as manifested in higher notched strength and fracture energy as well as significantly greater amounts of fiber pullout (Figure 2). The approach should be effective for fabricating coated fiber composites with virtually any architecture and configuration, circumventing current limitations in coating fabrics.

The low interlaminar tensile (ILT) strength of ceramic composites with two-dimensional fiber architectures presents serious challenges in the design of thermostructural components. Thus, in a parallel activity, the efficacy of 3D fiber architectures in oxide composites has been investigated. An illustrative example (Figure 3) shows the diffuse nature of matrix damage and the notch-insensitivity of strength in edge-notched bending tests of a composite with only 3% through-thickness reinforcements. In the absence of the latter, large scale delaminations develop at significantly reduced stresses.

Alternative protocols for measuring out of plane properties via interlaminar tensile (ILT) tests remain contentious because of the presence of free edges. One objective of a recent study was to introduce a modified test that eliminates machined edges and associated flaws from the volume that experiences stress, thereby enabling an ILT measurement more representative of the delamination response expected in actual components[2]. These goals were addressed by exploring oversized circular specimens. Experiments and numerical analyses of both the oversized and the standard configurations have been used to characterize the stress distributions and the failure conditions (Figure 4). The numerical results indicate that stress concentrations arise from thermal expansion mismatch in the near-edge regions of the standard configuration and exacerbate the deleterious effects of machining flaws. The ILT strengths of the oversized specimens are significantly higher than those of the standard configuration (by 30–50%), consistent with the exclusion of machining flaws and the reduced stress concentration. Such tests will be used in studying the through-thickness properties of the 3D oxide composites.

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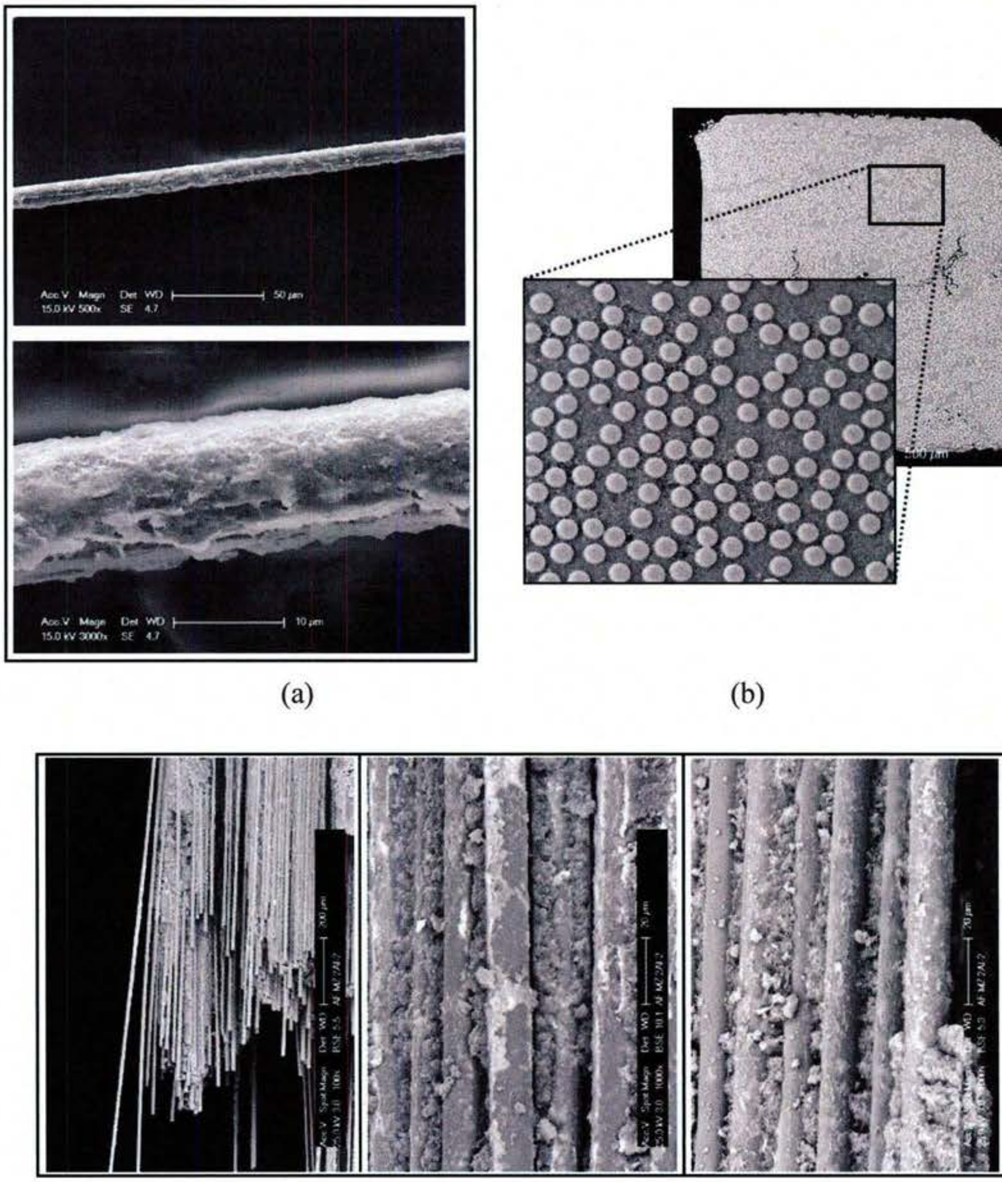
1. J.H. Weaver, J. Yang, C.G. Levi, F.W. Zok and J.B. Davis, "A Method of Coating Fibers in Oxide Composites", *J. Am. Ceram. Soc.*, in press.
2. J.H. Weaver, J. Yang, A.G. Evans and F.W. Zok, "A Modified Test for Measuring the Interlaminar Tensile Strength of Fiber-Reinforced Ceramic Composites, *Composite Sci. Tech.*, in press.



(c)

Figure 1 (a) Fibers with monazite coatings (provided by AFRL), (b) cross-section through a representative minicomposite specimen, and (c) fibrous tensile fracture surface.

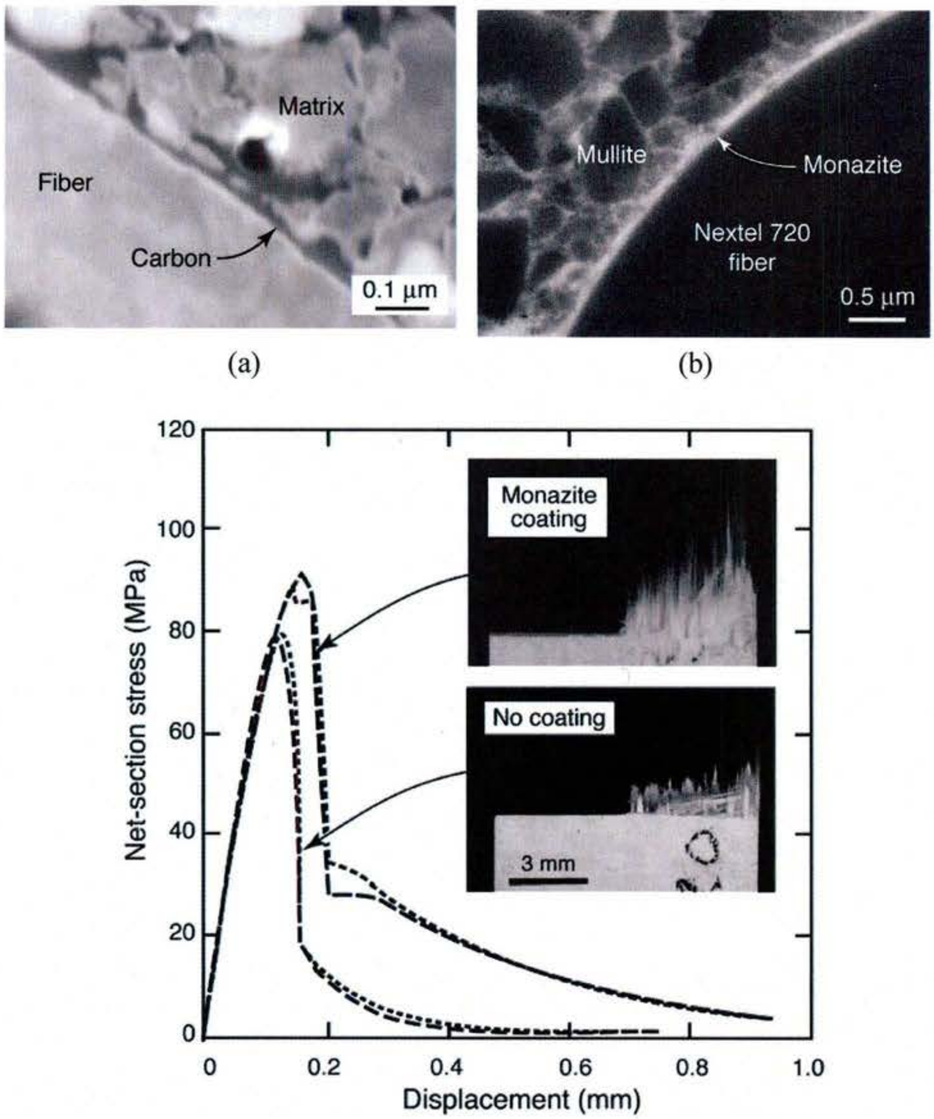


Figure 2 Cross-sections showing (a) carbon coating on fibers after matrix infiltration and firing and (b) monazite coating after pyrolysis of carbon and subsequent impregnation and firing of monazite precursor solution. (c) Demonstration of the superior tensile response of notched composite with monazite coating than that obtained without a coating.

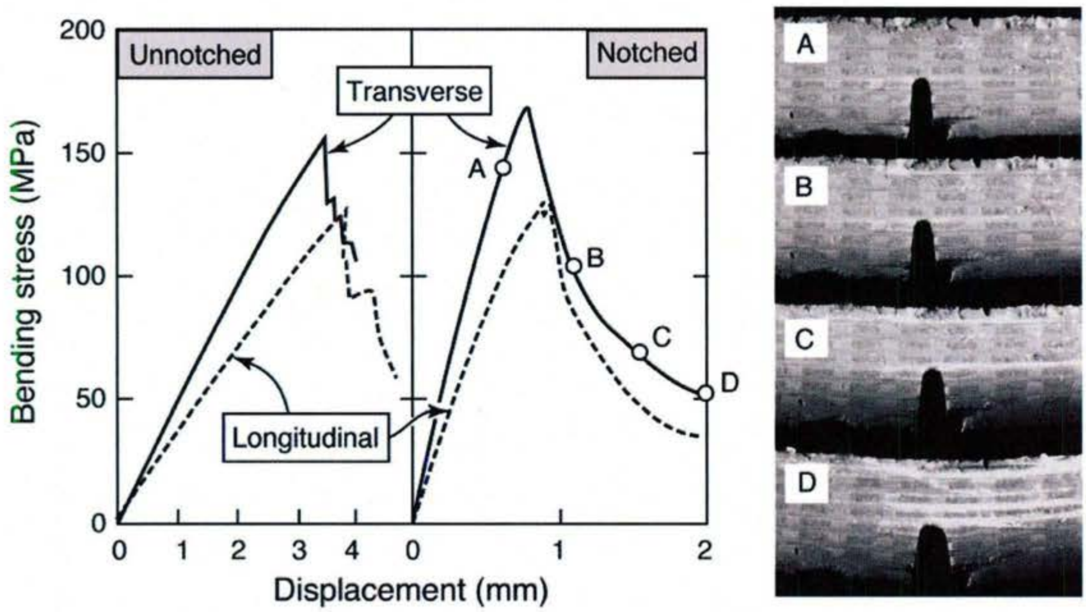
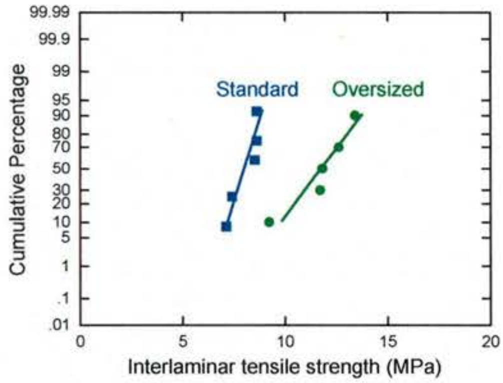


Figure 3 (a) Notch insensitivity of bend strength in the two principal in-plane orientations (denoted "transverse" and "longitudinal") and (b) diffuse damage progression in an edge-notched bend specimen of a 3D composite.



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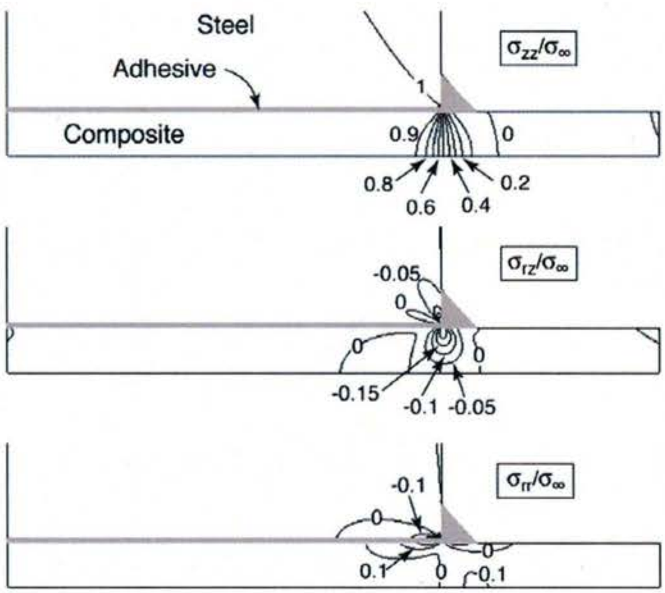

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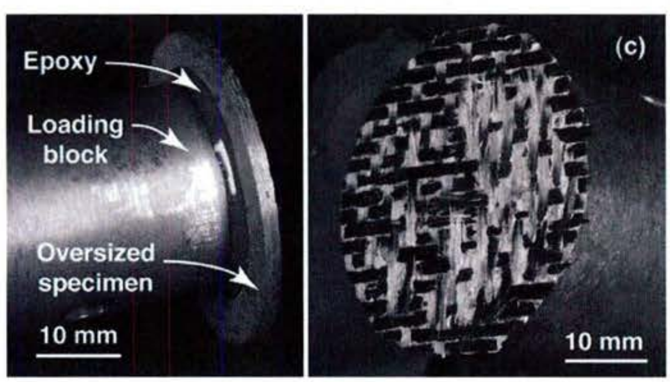

(c)

Figure 4 Effects of oversized specimens on the measured interlaminar tensile strength of a commercial composite.